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IS 2000 (Part 9) : 1989
(Reaffirmed 2001)

Indian Standard

CHEMICAL ANALYSIS OF BAUXITE

PART 9 DETERMINATION OF MAGNESIUM AND CALCIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

(First Revision)

भारतीय मानक

बॉक्साइट का रासायनिक विश्लेषण

भाग 9 परमाणु अवशोषण और स्पैक्ट्रमी प्रकाशमापी पद्धति द्वारा मैग्नीशियम और कैल्शियम ज्ञात करना

(पहला पुनरीक्षण)

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BUREAU OF INDIAN STANDARDS
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NEW DELHI 110002

April 1990 **Price Group 2**

Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee, MTD 29

FOREWORD

This Indian Standard (Part 9) (First Revision) was adopted by the Bureau of Indian Standards on 25 October 1989, after the draft finalized by the Methods of Chemical Analysis of Ores, Minerals and Refractories Sectional Committee had been approved by the Metallurgical Engineering Division Council.

IS 2000 : 1962 'Methods of chemical analysis of bauxite' covers the determination of different elements in bauxite. This standard was first published in 1962. The committee decided to revise this standard into different parts covering determination of each element in a separate part, which on publication, would supersede the determination of that element given in IS 2000 : 1962. This part covers determination of magnesium and calcium. The other parts are:

Part 1	Determination of loss on ignition
Part 2	Determination of silica
Part 3	Determination of alumina
Part 4	Determination of ferric oxide
Part 5	Determination of titania
Part 6	Determination of vanadium
Part 7	Determination of phosphorus pentoxide
Part 8	Determination of manganese

In this part oxalate method for determination of calcium oxide and oxine method for determination of magnesium oxide has been replaced by atomic absorption spectrophotometric method for determination of calcium and magnesium.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard
CHEMICAL ANALYSIS OF BAUXITE
PART 9 DETERMINATION OF MAGNESIUM AND CALCIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD
(First Revision)

1 SCOPE

1.1 This standard (Part 9) covers atomic absorption spectrophotometric method for determination of magnesium (in the range of 0·02 to 2·00 percent MgO) and calcium (in the range of 0·03 to 4·00 percent CaO) in bauxite.

2 REFERENCES

2.1 The following Indian Standards are necessary adjuncts to this standard:

IS No.	*Title*
IS 265 : 1987	Specification for hydrochloric acid (*third revision*)
IS 2000 (Part 1) : 1985	Methods of chemical analysis of bauxite: Part 1 Determination of loss on ignition (*first revision*)

3 SAMPLING

3.1 Samples shall be drawn and prepared as per the method prescribed in 2 of IS 2000 (Part 1) : 1985.

4 QUALITY OF REAGENTS

4.1 Unless specified otherwise, analytical grade reagents and distilled water shall be employed in the test.

5 DETERMINATION OF MAGNESIUM AND CALCIUM BY ATOMIC ABSORPTION SPECTROPHOTOMETRIC METHOD

5.1 Outline of the Method

Sample is fused with sodium carbonate and boric acid and dissolved in hydrochloric acid and hydrogen-peroxide. Suitable aliquots are aspirated into flame of atomic absorption spectrophotometer using nitrous oxide-acetylene burner for the determination of magnesium and calcium.

5.2 Reagents

5.2.1 *Sodium Carbonate*, anhydrous.

5.2.2 *Boric Acid*, solid.

5.2.3 *Concentrated Hydrochloric Acid* (*rd = 1·16*), (conforming to IS 265 : 1987).

5.2.4 *Hydrogen Peroxide*, 30 percent (*v/v*).

5.2.5 *Background Solution*

Weigh 5·72 g of super purity-aluminium turnings. Dissolve in 200 ml of NaOH solution (5 N). When the aluminium has dissolved, add 20·0 g of boric acid and 400 ml of concentrated hydrochloric acid. Transfer to a 1 000 ml volumetric flask, dilute to volume with water and mix. Store in a polyethylene bottle.

Dissolve 2·50 g of iron powder in 100 ml dilute hydrochloric acid (1 : 1). Warm to complete dissolution. Transfer to a 250 ml volumetric flask, dilute with water and mix. This solution contains 10 mg Fe/ml.

Dissolve 7·4 g of potassium titanium oxalate in 300 ml of water. Transfer to a 500 ml volumetric flask, dilute to volume with water and mix. This solution contains 2 mg Ti/ml.

In a stainless steel beaker, dissolve 1·392 g of silicic acid in 50 ml of NaOH solution (5 N). Warm if necessary to complete dissolution. Transfer to a 500 ml polyethylene volumetric flask, dilute to volume with water and mix. Store in a polyethylene bottle. This solution contains 1 mg Si/ml.

Dissolve 26·6 g of lanthanum chloride heptahydrate ($LaCl_2.7H_2O$) in water, dilute to 100 ml and mix. This solution contains 100 mg of La/ml.

Into each of the 250 ml volumetric flasks set aside for the preparation of the calibration solutions, pipette the following:

50 ml of aluminium-boric acid solution
10 ml of iron solution
10 ml of titania solution
10 ml of silica solution
50 ml of lanthanum solution

5.2.6 *Standard Magnesium Solution*, (100 and 10 mg of magnesium per litre).

Dissolve 0·250 g of oxide-free magnesium metal by slowly adding 75 ml of dilute hydrochloric acid (1 : 3). When dissolved, transfer to a 250 ml volumetric flask, dilute to volume with water and mix. Store in polyethylene bottle. This solution contains 1 000 mg of magnesium per litre. Transfer 25 ml of this solution to a 250 ml volumetric flask, dilute with water and mix. Transfer into a polyethylene bottle. This solution contains 100 mg magnesium per litre (A).

Transfer 25 ml of solution (A) to a 250 ml volumetric flask, dilute with water and mix. Transfer into a polyethylene bottle. This solution contains 10 mg magnesium per litre.

5.2.7 *Standard Calcium Solution*, (100 and 10 mg of Ca/litre),

Weigh 0·624 g of pure calcium carbonate in a 250 ml beaker, add 25 ml of water and 10 ml of concentrated hydrochloric acid. When the salt has dissolved, transfer in 250 ml volumetric flask, dilute with water and mix. Store in a polyethylene bottle. This solution contains 1 000 mg Ca per litre. Transfer 25 ml of this solution in a 250 ml volumetric flask and dilute. Transfer into a polyethylene bottle. This solution contains 100 mg Ca per litre (B).

Transfer 25 ml of solution (B) to 250 ml volumetric flask. Dilute with water and mix. Transfer it to a polyethylene bottle. This solution contains 10 mg Ca per litre.

5.3 Procedure

5.3.1 Weigh 1 g of sample (*see* **3.1**) in a platinum crucible. Add 3 5 g of sodium carbonate-boric acid fusion mixture (2 : 1) and mix thoroughly by stirring with platinum rod. Place the crucible in a muffle furnace at 800°C and immediately raise the temperature to 1 000°C. Keep the crucible in the furnace for 1 hour. Cool the crucible.

5.3.2 Strike the bottom of the crucible with the wooden handle of a spatula to crack the melt. Dislodge the solids by hitting the sides of the crucible with the wooden handle of the spatula and by gently pushing in the bottom and the sides. Fill the crucible with water and gently heat on a hot plate until the melt has softened. Transfer the mass to a 250 ml beaker using a gentle hot water jet. Add to the crucible a few ml of hot water, 5 ml of concentrated hydrochloric acid and 2 ml of hydrogen peroxide.

Gently heat on a hot plate until the residue is well dissolved. Remove the last traces of melt adhering to the sides of the crucible by adding 10 ml of water and 15 ml of concentrated hydrochloric acid. Heat the crucible on the edge of a hot plate until the solids have dissolved. Transfer the contents of the crucible to the 250 ml beaker. Add 2 ml of hydrogen peroxide.

Heat the contents of the beaker gently on the edge of the hot plate until the evolution of small bubbles has stopped.

NOTE — Do not heat strongly (silica will be precipitated). Continue heating if necessary until all the melt is dissolved.

Transfer the contents of the beaker to a 250 ml volumetric flask, dilute with water and mix. Transfer 25 ml of the solution into a 50 ml volumetric flask. Dilute to volume with water and mix. (Final test solution).

NOTE — For ores having higher magnesium or calcium contents than 0·25 percent MgO or 0·84 percent CaO respectively, an intermediate dilution step prior to the 25 ml/50 ml final dilution is required (for example 1/5 or 1/2·5).

5.3.3 *Blank*

Prepare the reagent blank using 3·5 g of fusion mixture and following the same procedure as specified in **5.3.1** and **5.3.2**.

5.3.4 *Calibration Solution* (*Magnesium and Calcium*)

From the standard magnesium solution (**5.2.6**) and standard calcium solution (**5.2.7**) prepare the calibration solution as follows:

To 130 ml (50 + 10 + 10 + 10 + 50 ml) of background solution (**5.2.5**) add the volume of respective standard solution given in Table 1. Dilute to 250 ml in volumetric flasks.

Table 1 Standard Solutions (Magnesium and Calcium) for Calibration Curve

(*Clauses* 5.3.4 *and* 5.3.7.3)

Percent MgO	Volume in ml of Standard Solution 10 mg Mg/L	Volume in ml of Standard Solution 100 mg Mg/L	Percent CaO	Volume in ml of Standard Solution 10 mg Ca/L	Volume in ml of Standard Solution 100 mg Ca/L
(1)	(2)	(3)	(4)	(5)	(6)
0·006 6	4·0	0	0·014	10·0	0
0·013	8·0	0	0·028	20·0	0
0·033	0	2·0	0·070	0	5·0
0·066	0	4·0	0·140	0	10·0
0·100	0	6·0	0·210	0	15·0
0·133	0	8·0	0·280	0	20·0
0·200	0	15·0	0·560	0	40·0
			0·840	0	60·0

Transfer 25 ml of each of the solutions to dry 50 ml volumetric flasks. Dilute to volume with water and mix. These solutions are the final calibration solutions.

NOTE — The range of magnesium or calcium that can be covered may vary from instrument to instrument. More diluted or more concentrated calibration solutions can be used by adjusting accordingly the portions of standard solutions required.

5.3.5 *Zero Calibration Solution*

Transfer 130 ml (50 + 10 + 10 + 10 + 50 ml) of background solution (**5.2.5**) into 250 ml volumetric flasks, dilute to volume and mix. Transfer 25 ml of this solution into 50 ml volumetric flask, dilute and mix.

5.3.6 *Adjustment of atomic absorption spectrometer*

Fit the burner in accordance with manufacturer's instructions. Light nitrous oxide — acetylene flame. Set the wavelength for magnesium (285·2 nm) and calcium (422·7 nm). After 10 minutes preheating of the burner, adjust fuel and burner to obtain the maximum absorbance. Aspirate water and calibration solution. to establish that the absorbance reading is not drifting and then set the initial reading for water to zero absorbance.

5.3.7 *Atomic Absorption Measurements*

5.3.7.1 Aspirate the calibration solutions and the final test solutions in order of increasing absorption starting with diluted blank test solution and the zero calibration solution, with the final test solutions being aspirated at the proper point in the series. Aspirate water between each solution and record the readings when stable responses are obtained. Repeat the measurements twice and average the readings.

5.3.7.2 Obtain the net absorbance of each calibration solution by subtracting the absorbance of the zero calibration solution (*see* **5.3.5**). Similarly, obtain the net absorbance of the final test solutions by subtracting the absorbance of the diluted blank test solution (*see* **5.3.3**).

5.3.7.3 Prepare a calibration curve by plotting the net absorbance values of the calibration solutions against the concentrations of magnesium or calcium, expressed as percent MgO or percent CaO (*see* **5.3.4** Table 1).

5.3.7.4 Convert the net absorbance values of the final test solutions to percent MgO or percent CaO by means of the calibration curves.

5.3.8 *Calculation of magnesium or calcium content*

The magnesium or calcium content, as a percentage by mass, is calculated from the following formula:

$$\frac{c \times 1{\cdot}000\,0 \times f}{m}$$

where

c = concentration, expressed as percent MgO or percent CaO, in 1·000 0 g test portion;

m = exact mass, in grams, of test portion; and

f = dilution factor.

If any further dilution is required then use the dilutions described.

5.3.8.1 From the calcium and magnesium content calculated above, the percentage of magnesium oxide and calcium oxide is calculated as follows:

$$\%\,\mathrm{Mg} \times 1{\cdot}67 = \%\,\mathrm{MgO}$$

$$\%\,\mathrm{Ca} \times 1{\cdot}39 = \%\,\mathrm{CaO}$$

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